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IS 7635 (1992): 2-Nitroaniline-Specification [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]



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“Knowledge is such a treasure which cannot be stolen”

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IS 7635 : 1992

(Reaffirmed 2002)

भारतीय मानक

2-नाइट्रोएनीलीन — विशिष्ट

(दूसरा पुनरीक्षण)

Indian Standard

2-NITROANILINE — SPECIFICATION

(*Second Revision*)

UDC 667.21 : 547.551.54

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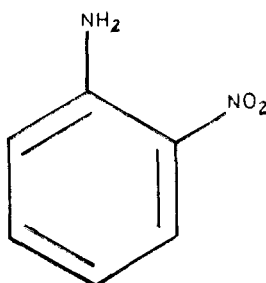
December 1992

Price Group 2

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Dry Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

2-Nitroaniline ($C_6H_6N_2O_2$) is an intermediate used in the manufacture of dyestuffs and pigments. It is also used as a diazo component in azoic dyeing in which case it is known as fast orange GR base. It is also known as 2-nitro-1-aminobenzene. It has the following structural formula:



(Molecular Mass 138)

C. A. S. Registration No. [88-74-4]

2-NITROANILINE

This standard was issued in 1975. The committee responsible for the preparation decided to revise it in order to incorporate the requirements as per the improved quality of the material being produced in the country. In the present version the requirements of assay and crystallizing point have been updated and the requirement of impurities (3-Nitroaniline and 4-Nitroaniline content) have been incorporated.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

2-NITROANILINE — SPECIFICATION

(Second Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for 2-Nitroaniline.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS No.	Title
1070 : 1992	Reagent grade water — Specification (<i>third revision</i>)
1997 : 1982	Burettes (<i>second revision</i>)
2552 : 1939	Steel drums (galvanized and ungalvanized) (<i>third revision</i>)
5299 : 1969	Methods of sampling and tests for dye intermediates

3 REQUIREMENTS

3.1 Description

The material shall be in the form of orange to golden yellow crystalline needles.

3.2 The material shall be slightly soluble in water and soluble in alcohol and ether.

3.3 The material shall also comply with the requirements given in Table 1.

Table 1 Requirement for 2-Nitroaniline

Sl No.	Characteristic	Requirement	Method of Test, Ref to
(1)	(2)	(3)	(4)
i)	Crystallizing point, °C, <i>Min</i>	69.0	7.1.2 of IS 5299 : 1969
ii)	Assay, percent by mass, <i>Min</i>	98.5	Annex A
iii)	3-Nitroniline content, percent by mass, <i>Max</i>	0.2	Annex B
iv)	4-Nitroanyline content, percent by mass, <i>Max</i>	0.2	Annex B

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in steel drums (*see* IS 2552 : 1989) lined with suitable

polyethylene film, or as agreed to between the purchaser and the supplier.

4.2 Marking

Each container shall bear legibly and indelibly the following information:

- Name of the material;
- Indication of the source of manufacture;
- Gross and net mass;
- Batch number; and
- The minimum cautionary notice worded as under:

‘DANGER ! HAZARDOUS SOLID AND
VAPOURS RAPIDLY ABSORBED
THROUGH SKIN’

4.2.1 The containers may also be marked with the Standard Mark.

5 SAMPLING

5.1 Representative samples of the material shall be drawn as prescribed in 3 of IS 5299 : 1969.

5.2 Number of Tests

Crystallizing point, assay and impurities of each individual sample shall be determined separately.

5.3 Criteria for Conformity

The lot shall be declared as conforming to the standard if the test results as obtained in 5.2 satisfy the corresponding requirements given in Table 1.

6 TEST METHODS

6.1 Tests shall be conducted according to methods prescribed in col 4 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise ‘pure chemicals’ and distilled water (*see* IS 1070 : 1992) shall be employed in tests.

NOTE — ‘Pure chemicals’ shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[Table 1, Item (ii)]

METHOD OF DETERMINATION OF ASSAY

A-1 OUTLINE OF THE METHOD

A known quantity of the sample is titrated with sodium nitrite solution in acid medium. The amount of sodium nitrite solution consumed may be expressed in terms of the amount of the amine present.

A-2 REAGENTS

A-2.1 Sodium Nitrite Solution — 0.1 N, freshly standardized against pure sulphanilic acid.

A-2.2 Concentrated Hydrochloric Acid — 30 percent (*m/v*).

A-2.3 Starch Iodide Test Papers

A-2.4 Potassium Bromide Solution — 25 percent (*m/v*).

A-3 APPARATUS

A-3.1 Mechanical Stirrer

A-3.2 Burette — see IS 1997 : 1982.

A-3.3 Beakers

A-3.4 Thermometer — Suitable to measure temperature in the range of 0 to 5°C.

A-4 PROCEDURE

Weigh accurately about 1 g of the sample and

transfer to a 1 000-ml beaker. Dissolve in 500 ml of water and 50 ml of a concentrated hydrochloric acid and heat, if necessary. Add crushed ice into the beaker and stir the mixture mechanically until the temperature is about 0 to 5°C. While stirring mechanically add the solution of standard sodium nitrite from the burette as rapidly as the spot test permits keeping the temperature below 5°C. Test the solution by spotting on starch iodide paper. If the rate of diazotization is slow, add a few ml of potassium bromide solution to obtain sharper end point. The end point is reached if an immediate faint blue coloured ring appears when the solution is spotted on starch iodide paper and which persists for a period of 5 minutes or so without further addition of nitrite solution.

A-5 CALCULATION

$$\text{Assay, percent by mass} = \frac{V \times N \times 13.8}{M}$$

where

V = volume, in ml, of standard sodium nitrite solution used;

N = normality of the sodium nitrite solution; and

M = mass, in g, of the sample taken for test.

ANNEX B

[Table 1, Item (ii) and (iv)]

DETERMINATION OF 3-NITROANILINE AND 4-NITROANILINE

B-1 OUTLINE OF THE METHOD

3-Nitroaniline and 4-nitroaniline are determined by thin layer chromatography.

B-2 REAGENTS

B-2.1 3-Nitroaniline Pure (Reference)	} Prepare 0.004 percent, 0.008 percent, 0.02 percent solutions of each 3-nitroaniline and 4-nitroaniline in methanol.
B-2.2 4-Nitroaniline Pure (Reference)	

B-2.3 Eluent

Benzene : ethylacetate (96 : 4)

B-2.4 Nitrous Fumes Chamber

A glass chamber of size 250 mm × 250 mm with a beaker of capacity 100-ml containing approximately 50 ml of concentrated HCl (30 percent *m/v*). The nitrous fumes are generated by adding a little solid sodium nitrite everytime before inserting the plate.

B-2.5 Spray Reagent

0.1 percent (*m/v*) solution of American Base (N-1-Naphthylethylene diaminedihydrochloride) in methanol.

B-3 APPARATUS

B-3.1 Thin Layer Chromatographic Chamber — 250 mm × 100 mm × 250 mm.

B-3.2 Micropipette of 10 Microlitre Capacity**B-3.3 Pre-coated Thin Layer Chromatography Plates**

TLC plates (200 mm × 200 mm) coated with silica gel, about 0.25 mm thickness and with a fluorescence of F-254 nm.

B-4 PROCEDURE

B-4.1 Weigh accurately about 4 g of the sample (100 percent basis) and dissolve in 100 ml, methanol. Take a plate coated with silica gel and spot 10 litres of each of the test solution and reference solutions along the start line at a distance of 3 cm from one another. Saturate the chromatographic chamber with eluent. Insert the plate in the chamber and allow to run for

about 25-30 minutes during which time the solvent front travels approximately 10 cm past the start line. Remove the plate from the chamber and dry in air. After drying put the plate in the nitrous fumes chamber for about 30 seconds. Remove the excess nitrous fumes by blowing air on it. Spray the plate with American base (**B-2.5**) when the spots (red violet) characteristics of 3-nitroaniline and 4-nitroaniline become visible at *R_f* 0.43 and 0.29 respectively.

B-4.2 Reporting

Estimate the quantities of the impurities by comparing the colour intensity of the spots of the material under test with those of the reference samples.

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